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Report No. SA-TRI8-1097

SPUTTERING INTERMEDIATES ON TITANIUM
TO IMPROVE ADHESION OF ELECTRODEPOSITED CHROMIUM

Technical Report

Hassion, Dr. Francis X.

Date 19 April 1966

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SPUTTERING INTERMEDIATES ON TITANIUM TO IMPROVE ADHESION
OF ELECTRODEPOSITED CHROMIUM

Technical Report

Hassion, Dr. Francis X.

PROJECT TITLE: Manufacturing Chemistry, Erosion- and Corrosion-Resistant
Coatings

PRON: M1-3-23043

Preparing Agency: Springfield Armory, Springfield, Massachusetts

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FOREWORD

This is a summary report of work done on Contract DA-069-AMC-467(W) by R. T. Luedeman and T. Matley at Weston Instruments and Electronics Division of Daystrom Corporation. The work described herein was done by the above authors under the technical supervision of Springfield Armory Research and Engineering Division, Support Research Branch, and is supplemented by technical comments and visual presentation of the results provided by Dr. F. X. Hassion of the Springfield Armory Research and Engineering Division, Support Research Branch.

ABSTRACT

Intermediate layers of chromium, nickel, and/or iron have been sputtered on titanium substrates. In the latter cases, upon subsequent chromium electrodeposition, adherence was localized and nonuniform. Chromium electrodeposits on chromium intermediate-sputtered layers, however, were uniform and adherent as indicated by bend tests. Experimental procedures are described and results are discussed.

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SUBJECT

Sputtered Intermediates on Titanium

OBJECTIVE

The purpose of this research was to achieve sputtered intermediates on titanium substrates to improve adhesion of electrodeposited chromium.

SUMMARY OF CONCLUSIONS

Iron, nickel, and/or chromium have been sputtered on titanium substrates. Uniform and adherent chromium electrodeposits have been successfully applied to the chromium intermediate, but have not been applied to the iron or nickel intermediate layers.

RECOMMENDATIONS

The results obtained in the work described herein show that further study of the sputtering method is definitely warranted.

The study should be aimed at (1) establishing suitable criteria for adhesion, (2) refining the equipment and process used, and (3) standardizing the process for ultimate production use.

1. INTRODUCTION

Because titanium is a lightweight metal with high strength properties and a fairly high melting point (1800°C), it has received considerable attention with regard to its use in the manufacture of advanced weapons and weapons system components. One objection to its use stems from its affinity for oxygen, which results in surface layers of titania. The ease of formation and the tenacity of the contaminating surface layers have made it almost impossible, up to now, to obtain completely adherent electrodeposited coatings which would offer consistent protection at elevated temperatures. Much work has been done in the past to develop methods of applying hard, wear-and-temperature-resistant coatings, such as chromium-to-titanium surfaces, with varying degrees of success. The investigations described in this report are a further step in that direction, and deal with a means of applying an adherent intermediate layer on the titanium substrate, upon which an electrodeposit of chromium could then be uniformly and adherently applied. The method used to obtain such an intermediate layer was sputtering, a technique described qualitatively below.

In the sputtering process, two electrodes are placed in a controlled low-pressure atmosphere of an inert gas such as argon. Under appropriate voltage conditions, this technique removes material from the surface of a cathode. Since the immediate aim is to clean the titanium substrate sufficiently to receive an adherent layer of material, the surface is first made to be the cathode. A high voltage ionizes the ambient gas, and the positive ions bombard the cathode; this dislodges oxygen and other "impurities" which are then either pumped out of the system or "getterred" by the anode. Upon completion of the cleaning phase, the intermediate layer material (cathode disc) becomes the cathode and the cleaned titanium now acts as either an anode or an unshielded floating element. The cathodic bombardment creates a partial pressure of intermediate layer material, which then deposits on the titanium surface. Generally speaking, a high voltage source, approximately 5000 volts, may be required, with a power dissipation amounting to about 100 watts. The precise details of what occurs in the plasma of a glow discharge, whether the removal is atomic or molecular, is not important here since the results are definite, controllable, and reproducible. The bibliography includes a number of publications which deal with the theoretical aspects of the process.

2. EXPERIMENTAL PROCEDURES

Four-inch diameter cathode discs, one-quarter inch thick, were used for sputtering; these included iron, nickel, and chromium. Commercially pure titanium (Crucible Steel Corporation, alloy A-70, equivalent to Titanium Metals Corporation Ti 75a) was sheared into 1 inch by 3 inch panels, a size practical for the sputtering and electroplating operations. Figure 1 shows the stock titanium.

2. EXPERIMENTAL PROCEDURES - Continued

In the chemical cleaning of titanium, a panel was degreased by washing with C.P. acetone and allowed to dry. The panel was next cleaned in acid solution of the following composition: Water, 10cc; HNO_3 (conc.), 22 cc; and HF (conc.), 5 to 10 cc. Approximately $1/2$ of the volume of acid mix was used to cover the panel and approximately another $1/3$ of the volume of acid mix was added when the action slowed down. This latter step was repeated. The acid, diluted with water, was poured off. The panel was washed with detergent solution and subsequently rinsed with deionized or distilled water. The panel was then rinsed with denatured alcohol, rinsed with C.P. acetone and allowed to dry. Water marks or discolorations on the panel were unacceptable. Figures 2 and 7 illustrate good and poor chemical cleaning of titanium, respectively.

Sputter-cleaning of the iron cathode was accomplished at 1000 volts and 80 milliamperes in one-half hour or less. A lower wattage was used in cleaning the titanium so that the titanium would not become too hot. The time to sputter-clean the titanium varied from one-half hour to several hours depending upon its condition and other variables that affected the efficiency of the process. The titanium was cleaned at 2000 volts and 20 to 30 milliamperes. Approximately 0.8 micron thickness of iron deposit per hour was obtained in sputtering. Sputter-cleaning was required to achieve adhesion. Figures 3 and 8 illustrate good and poor sputter-cleaning of titanium, respectively.

In chromium sputter-cleaning, the chromium disc was connected electrically as the cathode and an aluminum shield was used as the anode. The flow of argon was controlled so that a potential of 1500 volts resulted in a current flow of 90 milliamperes. The sputter-cleaning of chromium normally required about 30 minutes. While the chromium was being cleaned, the aluminum shield prevented the deposition of chromium on the titanium sample. The chromium disc was also protected by an aluminum anode shield in the sputter-cleaning of titanium. Sputtering of chromium was accomplished at 1500 volts and 90 milliamperes. It was calculated, by use of the weight deposited on the titanium, that a layer of chromium of 1.5 microns thickness was obtained in one hour. Figures 4 and 9 illustrate good and poor sputtered intermediates, respectively.

In the plating work, a 250 gram per liter chromate bath of 100 to 1 sulphate ratio operating at 55°C was used. The best results were obtained with a 1 inch by 3 inch anode (6 per cent antimonial lead) turned 90 degrees

2. EXPERIMENTAL PROCEDURES - Continued

so that an edge was presented to the face of the 1 inch by 3 inch cathode (the back of which was masked with 3M Minnesota Mining and Manufacturing Company, No. 271 tape). In effect, there was an anode-to-cathode surface area ratio of 2:1. The electrode separation distance varied from 1 inch to 1/4 inch. A reverse etch was applied for 15 seconds at 4 amperes total, and plating proceeded immediately at 6 to 8 amperes total (approximately 6 volts). Plating times of 20 to 60 minutes gave electroplated thicknesses of 1 to 3 mils which is greater than normally obtained from standard plating baths. Based on the weight of chromium removed with the use of two different baths, it appeared that a reverse etch at IASI can be tolerated up to 1 minute before removal of a weight equivalent to 1 micron of thickness. Attempts were made to arrange the electrodes to obtain uniform plate with the expectation that etching would also tend to be more uniform.

Initial adhesion tests were conducted by the use of a strongly adherent tape. Later, bending was used in conjunction with the tape test.

3. RESULTS

The weight of sputtered deposit versus time demonstrates that the rate is constant. Results indicate that the nickel sputtering rate is similar to that of iron. Chromium evidently sputters much faster than iron or nickel. It was assumed that the deposition rate is inversely proportional to the distance between the intermediate metal disc (cathode) and titanium substrate. Twice the separatory distance in the case of chromium would then give the same rate as found for nickel and/or iron. This was experimentally verified.

In a typical example of an iron intermediate sputtered on titanium, a zone of adherent electrodeposited chromium, arc-shaped, and approximately an inch long by a quarter inch wide, gave sufficient adhesion to survive a bend of approximately 4t radius. The back of the panel, having no iron, did not plate and other zones on the front had no plating or it was of such poor quality that the chromium flaked off easily. The difficulty encountered here was probably due to a nonuniformity of the activating reverse etch in the chromium electroplating bath which is the explanation offered by Luedeman and Matley and is inconsistent with the idea that iron presents an active surface. Possibly oxide coating formed in transfer from the sputtering chamber to the plating bath was a factor and this was not uniform. The reverse etch is especially important because nonuniform etching may completely remove the deposited intermediate from one area before another area is activated.

3. RESULTS - Continued

No specimen of electroplated chromium over iron or nickel intermediates exhibited more than 10 to 20 per cent adherent coverage; the remainder of the surface exhibited severe flaking. Iron is quite susceptible to chemical action. The unsatisfactory adhesion and the inadequate surface coverage of the chromium electrodeposits are probably related to this chemical action.

In the example of a chromium intermediate sputtered on titanium, total coverage of the chromium intermediate with a chromium electrodeposit was possible. Also the adhesion, as indicated by tape and bend tests (1t bend radius in some cases), appeared excellent. Figures 5 and 10 illustrate good and poor chromium electrodeposits.

Initially, a reverse etch was believed necessary in the chromium bath as a preparation of the sputtered intermediate before chromium electroplating. All samples in which iron and nickel intermediates were used failed to give adherent coverage with or without reverse etch in the chromium bath. It was noted that electroplating was more successful without the reverse etch which is contrary to standard chromium plating experience. Thus it was considered possible that the reverse etch could cause lifting and destruction of the sputtered intermediate even at such low levels of etching that very little intermediate layer should have been removed. The omission of the reverse etch, however, is only possible if the surface to be plated is resistant to contamination during the handling process.

Specimens illustrating both good and poor properties were selected at various stages of preparation of a titanium substrate with chromium intermediate and electrodeposit. These specimens are shown in Figures 1 through 11 (Appendix A). These figures, taken at 1 magnification, are captioned on page 7. Cross sections of titanium substrate covered with a chromium intermediate plus chromium electrodeposit are illustrated in Figures 12 and 13. These figures are photomicrographs which were taken in diallyl phthalate, polished metallographically, and etched in an aqueous solution containing 1 per cent HF and 2 per cent HNO_3 . The dark region behind the intermediate layer in Figure 13 may be indicative of hydrogen charging occurring in the electroplating process. This is noticeably absent in Figure 12 which represents a sample not electroplated.

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ILLUSTRATIONS

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| 2 | Good Chemical Cleaning |
| 3 | Good Sputter Cleaning |
| 4 | Good Sputtered Intermediate |
| 5 | Good Chromium Electroplate |
| 6 | Good Chromium Adhesion |
| 7 | Poor Chemical Cleaning |
| 8 | Poor Sputter Cleaning |
| 9 | Poor Sputtered Intermediate |
| 10 | Poor Chromium Electroplate |
| 11 | Poor Chromium Adhesion |
| 12 | Cr-Intermediate-Ti Cross Section |
| 13 | Cr-Cr-Intermediate-Ti Cross Section |

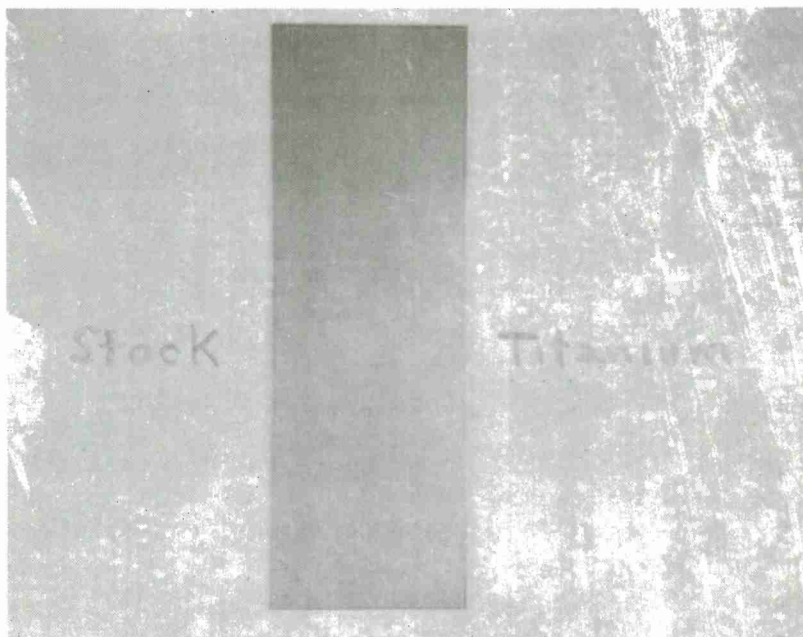


Figure 1. Stock Titanium

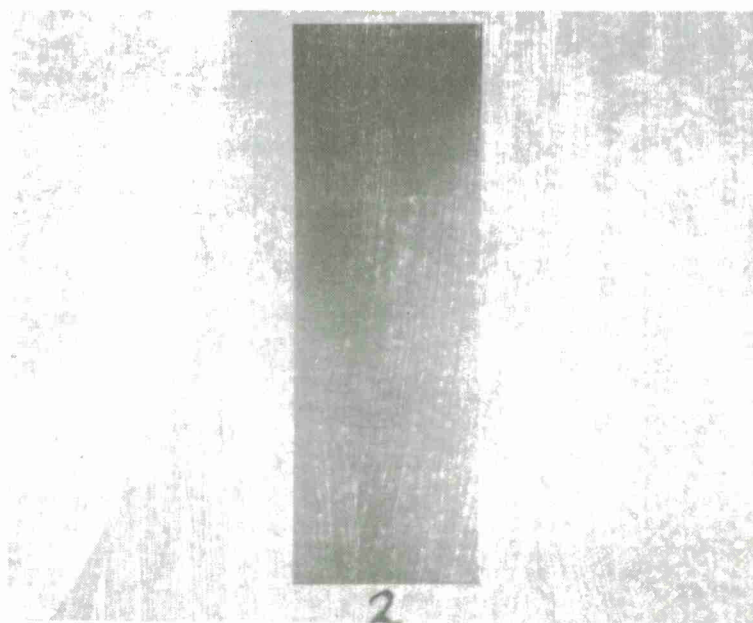


Figure 2. Good Chemical Cleaning

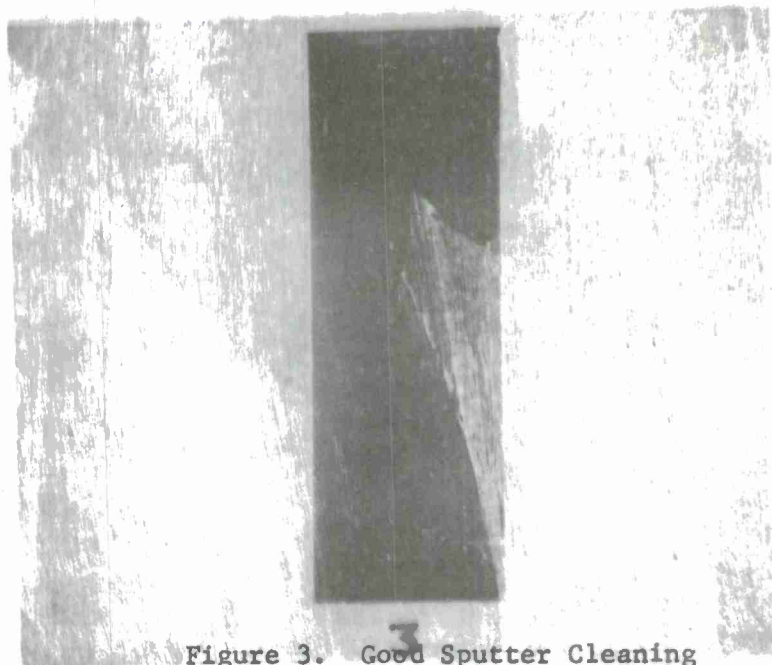


Figure 3. Good Sputter Cleaning

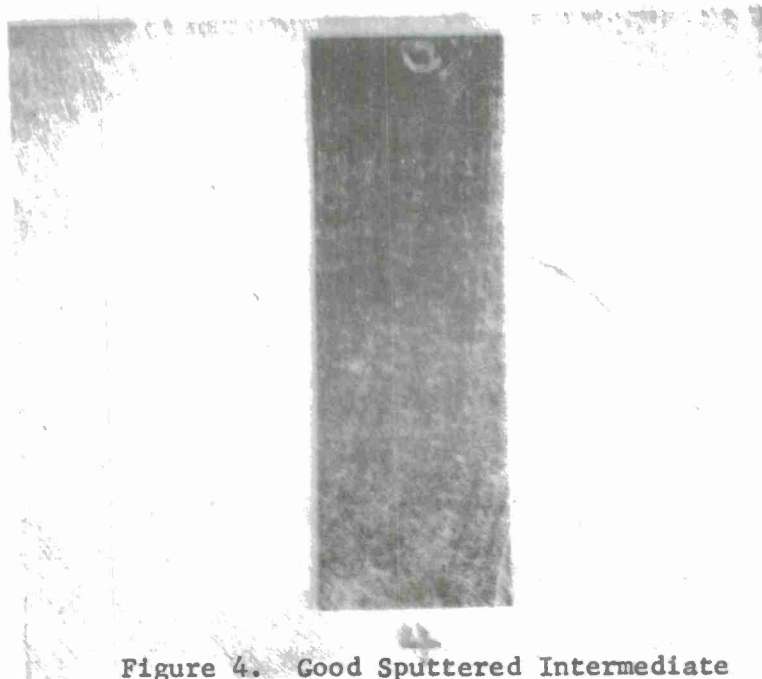


Figure 4. Good Sputtered Intermediate

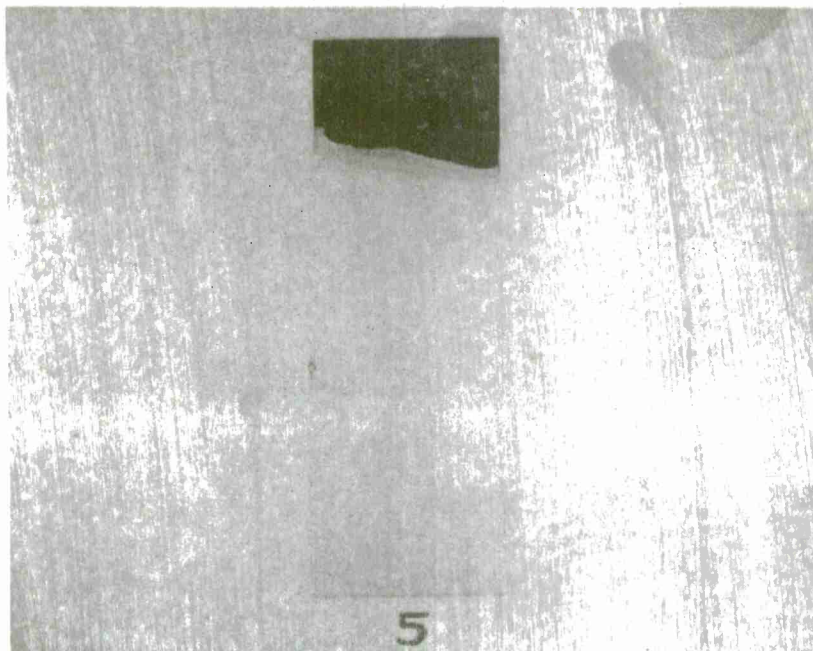


Figure 5. Good Chromium Electroplate

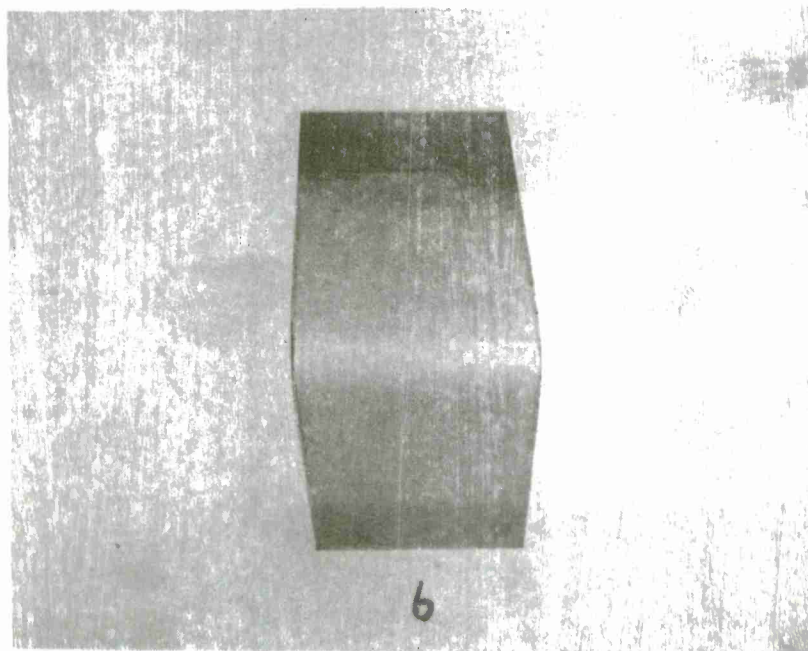


Figure 6. Good Chromium Adhesion

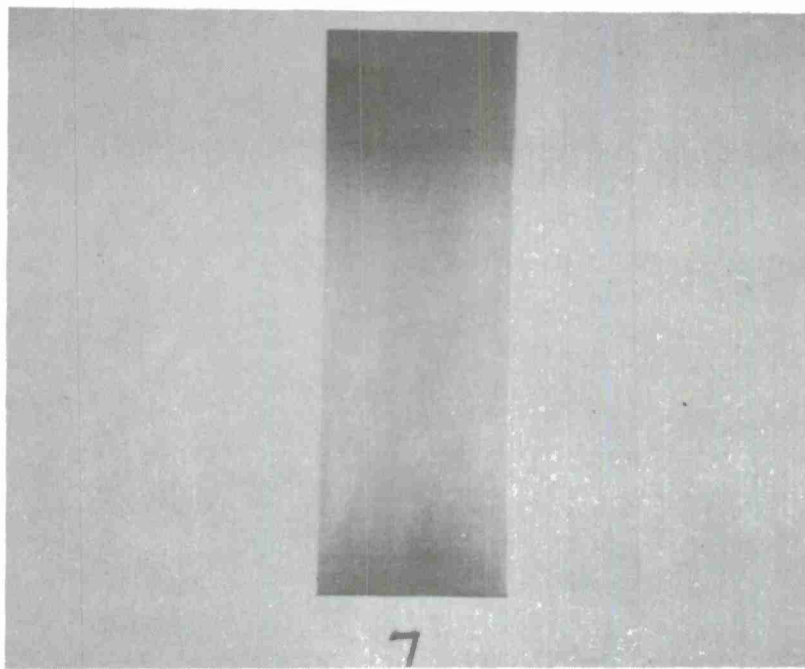


Figure 7. Poor Chemical Cleaning

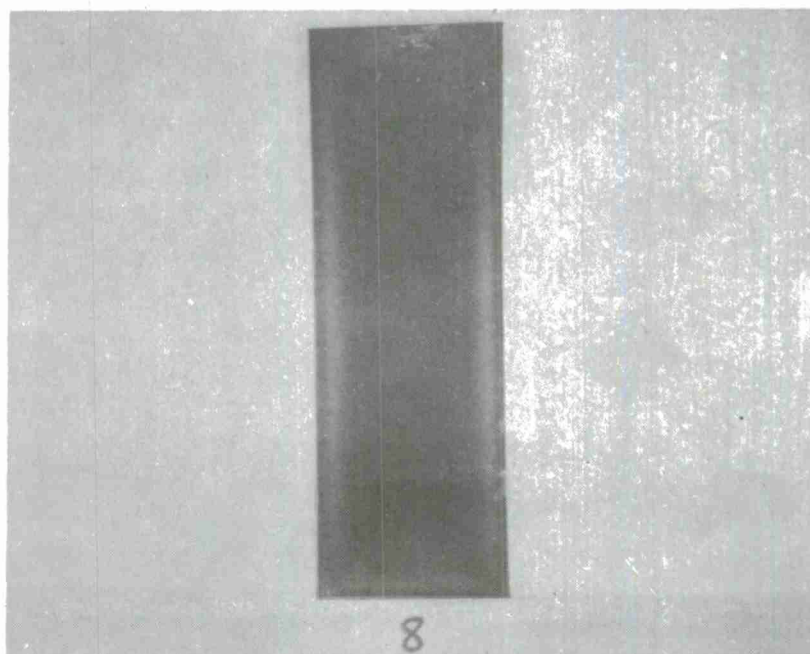


Figure 8. Poor Sputter Cleaning

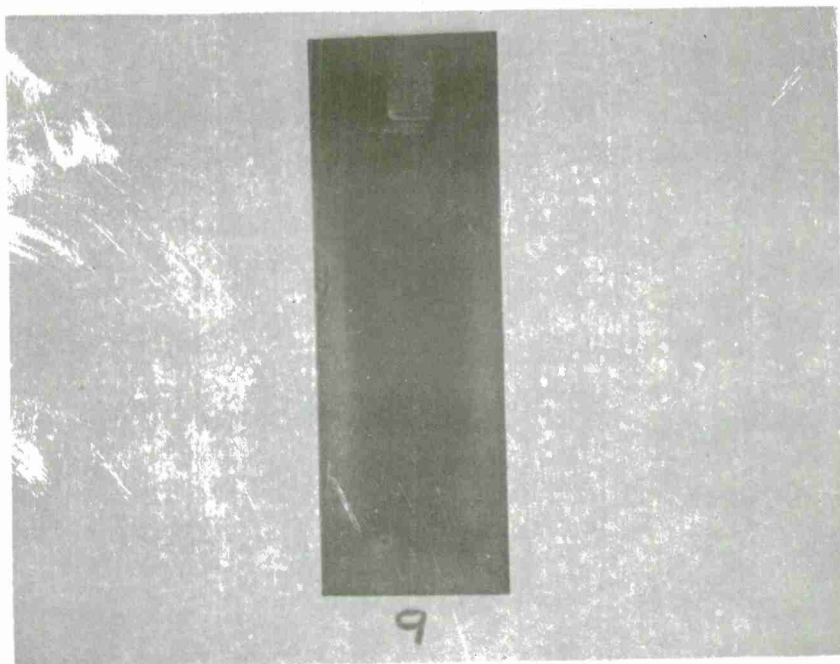


Figure 9. Poor Sputtered Intermediate

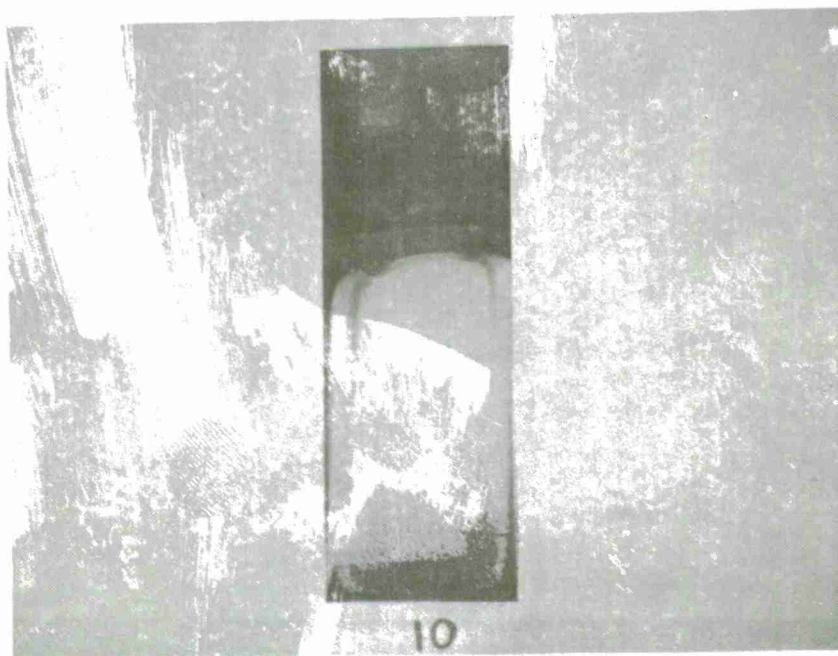


Figure 10. Poor Chromium Electroplate

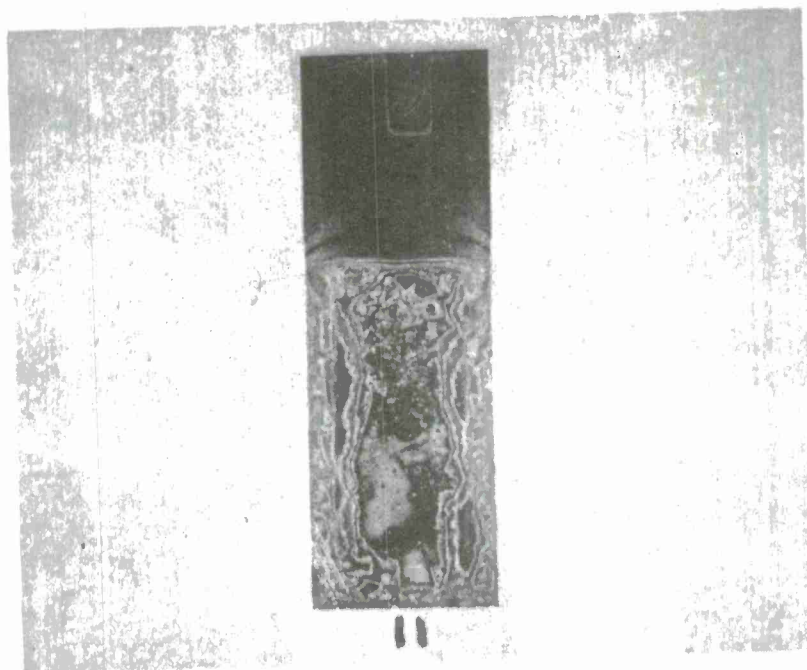


Figure 11. Poor Chromium Adhesion

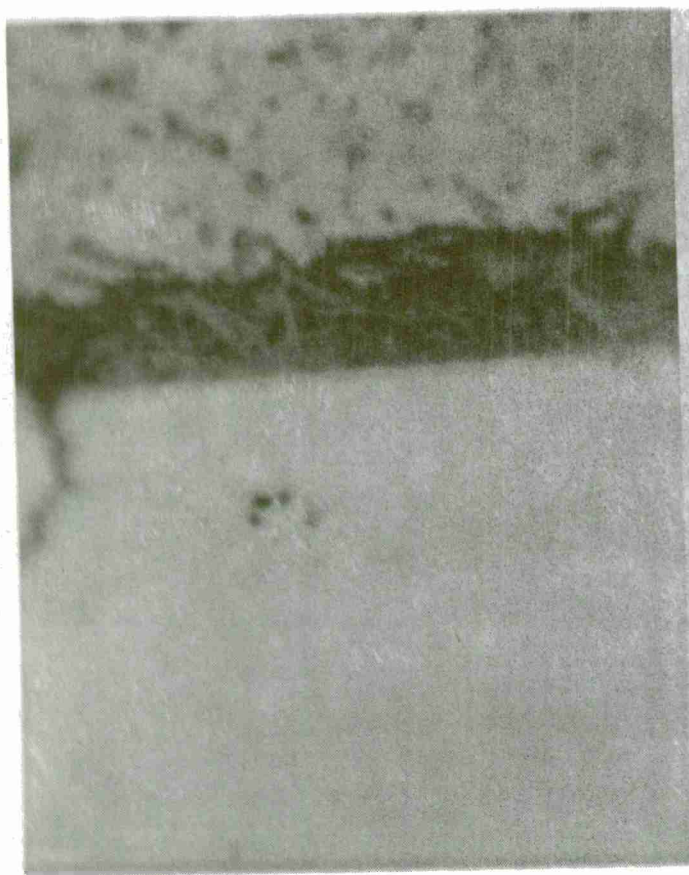


Figure 12

Cr-Intermediate-Ti
Cross Section



Figure 13

Cr-Cr-Intermediate-Ti
Cross Section

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13. ABSTRACT

Intermediate layers of chromium, nickel, and/or iron have been sputtered on titanium substrates. In the latter cases, upon subsequent chromium electrodeposition, adherence was localized and nonuniform. Chromium electrodeposits on chromium intermediate-sputtered layers, however, were uniform and adherent as indicated by bend tests. Experimental procedures are described and results are discussed.

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| 3. Composite materials | | | | | | |
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